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ARO-ERO PHOSPHORS FOR MARKING, CODING AND INFORMATION

STORAGE

4th and FINAL REPORT

October 2003

Contract No. DAAD19-02-1-0453

R+D 9350 EE01

Principal Investigators

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ARO-ERO PHOSPHORS FOR MARKING, CODING AND INFORMATION STORAGE

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Contract No. DAAD19-02-1-0453

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Introduction and background

This report describes the work carried out under the ARO-ERO contract No. DAAD19-02-1-0453, which began on the 1st September 2002. The objective of this preliminary study was to point to simple methods to improve marking and coding for combat identification to reduce the number of fratricide incidents among American and allied casualties incurred during recent conflicts.

From our previous studies the use of powder phosphors was identified to have the potential to meet some of these marking and coding requirements, especially in low light levels and poor visibility. The use of phosphors is widespread in modern technologies; they can be mass-produced and are in general widely available, no expensive equipment or control is needed to lay down large uniform layers of material. Large areas of phosphors can be laid down and covered in plastic sheeting etc. The recent availability of near

1

ultraviolet light emitting diodes in addition to infrared emitting systems made the remote excitation of phosphors a practical possibility.

In our initial proposal we listed two possible areas for investigation with the aim of producing practical devices to reduce fratricide incidents. These were:

1. Storage phosphors

2. Electroluminescent to Infrared emitting phosphors

A third possible area for investigation was agreed upon after the group had discussed the best approaches during the first 3 months as it became clear that the first two approaches might not lead to demonstrators within the time period of the project.

Our third approach involved the development of photoluminescent phosphors which could convert specific energies to specified infrared signals e.g.

(a) Visible to Infrared

(b) Ultraviolet to Infrared

(c) Infrared to Infrared (Stokes)

(d) Infrared to Visible (anti-Stokes)

In order to assess these possibilities we were granted a one year DARPA contract.

The proposal originally sought to combine the aforementioned light sources with storage, infrared emitting and anti-Stokes (upconverting) phosphors to produce specific marking and coding materials.

Initially on a very limited program we wished to establish with demonstrators that would point toward systems that would point to be used in the field of combat to eliminate the number of fratricides.

It was important at the start of the program to seek guidance to identify a number of points that would allow us to channel our investigations directly on U.S. Army requirements. Points such as the favoured excitation and emission wavelengths, would the priority of switching be conferred upon the target, what would be the preferred distances between source of excitation and the responding target.

We were directed at the start of the program that a night vision sight (NVS) should be obtained. From this being the only guidance received it was clear that some of the original objectives would not be applicable due to the imaging constraints of the NVS. The use of a NVS indicated that detection of a friendly signature would have to be observed in the infrared region to eliminate visible detection by the enemy. The suppression of visible emissions discounted the use of storage phosphors (infrared to visible) and anti-Stokes (infrared to visible).

An assumption was made that the enemy would also have access to NVS's. Although the NVS's available to the enemy would be 1st or early 2nd generation devices that do not have imaging capabilities at longer wavelengths in later generation scopes that are available to U.S. forces.

It should be emphasised that the first three months were spent familiarising ourselves with a number of new aspects that would allow us to proceed with the program in addition to assessing and obtaining new equipment and materials.

An extensive literature search was conducted to establish a baseline for the program. The search was very labour intensive, due to the fact that most of the papers on storage and infrared sensitive phosphors were published in the years just after the Second World War. Modern databases were not helpful in searching this far back in time therefore manual searches were undertaken at the British and Royal Society of Chemistry Libraries. Despite this problem the survey was very fruitful and enabled us to identify some areas of this subject and systems that had been abandoned for many years and were pertinent to this project.

Familiarisation with the Night Vision Sight (NVS) during the initial months after obtaining was a learning curve that allowed us to identify the infrared (IR) phosphor systems that are suitable for this program and eliminate those that are inadequate within the 'world view' observed through the NVS. It has also shown to us the problems associated with the use of NVS in the field, as to the effects of 'bloom' and the need for filters etc under certain conditions.

As the program progressed it was deemed sensible to discontinue work on finding an efficient EL infrared system because this would require a major long term study to achieve this goal. Also investigations into other systems that were initially candidates for infrared emitting materials such as $LiAl_5O_8:Fe^{3+}$ were abandoned because their infrared properties were not comparable in efficiency and wavelength range with the ZnCdS type systems. It was felt that it would be more rewarding by directing all our efforts towards the ZnCdS infrared emitting phosphors.

Night Vision Sight

Due to the sensitivity of the Pyser PNP-Hypergen NVS we had difficulty in observing pure IR emissions at the outset of the program. Due to our lack of familiarity with the technology and the world viewed through the scope it took us some time and effort to overcome these problems.

For instance viewing through the NVS, a phosphor and a white powder (under UV illumination), in addition to UV, optical and IR wavelength LEDs, all their emitted signatures are similar. Therefore we could not distinguish between emitting wavelengths.

The 'bloom' that is associated with NVS was also a problem, in the case of LEDs we had to decrease the intensity of the collecting aperture to very low levels to eliminate it. These problems were overcome by using a Long Wave Pass Filter (LWPF) attached to the front lens of the NVS to block optical and near infrared wavelengths below 900 nm. The LWPF had a chosen optical density of 4.0 (transmittance of 0.01%), although a low level of light is transmitted through the LWPF this would be adequate for the NVS to gather an optical image of the field of view. Under these conditions the pure IR emissions could be observed and allow the NVS to function by imaging the surrounding environment. With the optimisation of the NVS for our specialised use we were able to start assessing the phosphors we had synthesised and the particular benefits of each system.

Laser Induced Luminescence

The Laser Raman Facility here at Greenwich was able to give spectra of the infrared emitting phosphors. Laser induced luminescence

spectroscopy using helium-neon laser excitation of wavelength equal to 632.8 nm allowed us to observe the effect of various dopants and concentrations on emission band intensities and shifts across the IR region. Although only qualitative assumptions as to the emitted light intensity could be made. These spectra can be seen in Figures 1 to 3.

Infrared Emissive Systems

A number of phosphor candidates were investigated to ascertain their infrared emissive properties such as $LiAl_5O_8:Fe^{3+}$, ZnCdS:Cu,Mn (EL), ZnCdS:Cu (Mn and Ag) and storage phosphors. All phosphor systems investigated were found to have inferior infrared properties when compared to ZnCdS phosphors. Thus it was decided to concentrate on the ZnCdS phosphor system. This phosphor system had the advantage that it was excited by UV and blue light (432 nm), although the system could also be excited with deep red emitting source. It should be noted that we found no quantitative data on the efficiency of infrared emitting systems. The data was also very limited with regard to the range of the excitation spectra no record of phosphors responding to energies above 570 nm were found. It is therefore assumed much of our data is entirely new. The $Zn_{0.4}Cd_{0.6}S:Cu$ phosphor was found to have an emission peak in the infrared region tailing off beyond 900 nm although a peak at 750 nm was also present (Figure 1). The peak at 750 nm would therefore be detectable by all generations of NVS and would need to be suppressed. It was reasoned that by adjusting the Zn and Cd ratios in

addition to the use of different activators the 750 nm peak may be eliminated leaving only the peak at 900 nm to be observed.

From the laser induced luminescence spectra the emission peak is shown to be very wide tailing off into the infrared beyond the limits of our spectrometer (1000 nm). It should be noted that LED emissions in this region are very narrow compared to the emissions of our new infrared phosphors and this may have important implications in the applications of the new phosphors.

This led to the preparation of ZnCdS:Cu phosphors, by the sulphur/hydrazine hydrate and thiourea dioxide methods, with the following Zn:Cd ratios: 30:70, 20:80, and 10:90. It should be noted that the amount of dopant and the amount of sodium chloride used were not altered from the quantities used in the preparation of $Zn_{0.4}Cd_{0.6}S:Cu_{0.2}$ phosphor. This was done to limit the variables in the experiment to the Zn:Cd ratio. The Zn/Cd systems were then assessed with the NVS with the LWPF in place. It was observed that the $Zn_{0.3}Cd_{0.7}:Cu_{0.2}$ sample displayed the strongest IR emission with a 2% sodium chloride flux. The thiourea method also produced phosphors with stronger infrared emissions when viewed through the NVS.

It was found that the $Zn_{0.3}Cd_{0.7}:Cu$ phosphors infrared emission could be excited over a wide range of wavelengths. Infrared emissions were observed using the following excitation sources 254 and 366 nm mercury lamp, white light EL panel, fluorescent lamp, in addition to 370, 432, 590, 620 and 880 nm LED excitation. Over the range 370 to 620 nm the IR emission intensity appears to be similar (intensity judgements are subject to interpretation by the observer), whereas the IR exciting wavelengths give weaker IR emissions.

The addition of gold (AuCl$_3$) as a co-activator substantially improved the infrared emission. Indium as a co-activator was found to have the strongest infrared emission when viewed through the NVS. Using laser induced spectroscopy the infrared peak was observed to have the maximum proportion of its spectrum in the infrared region (Figure 2).

Further work was carried out with the Zn-Cd-S-Se system to examine the effect of partial replacement of sulphur by selenium. The results indicated so far on the samples synthesised that Zn-Cd-S-Se systems are inferior to the ZnCdS systems. Clearly much optimisation of these systems is required.

The main problem when evaluating the intensity of infrared emission was that it was purely qualitative and subjective. It was reasoned that the infrared emissions perceived through the NVS could be measured using an in-line photometer. This would allow a quantitative measurement of how each phosphor compared. Thus the infrared light output from a range of ZnCdS and ZnCdS/Se phosphor powders with a selection of activators was imaged through a night vision scope (NVS).

The NVS was mounted on a tripod one and a half metres from the surface of the phosphor sample. A 900 nm long wave pass filter (LWPF) was connected to the front lens of the NVS. The viewing end of the NVS was placed directly in front of a photometer. The sample was excited with 366 nm ultraviolet radiation and white light. The measurements were collected through a circular aperture on thick black card that was positioned in between the sample and the NVS. The card was used to obscure all the exciting radiation so that only a small area of infrared light was imaged by the NVS. Two aperture diameters were used (5 and 7 mm) measurements were also

taken without the card in place. The actual light measured was the infrared emission from the phosphor that was converted to green light by the NVS imaging system. Thus the measurements are a quantitative comparison of the infrared emissions.

The results are shown in Tables 1 and 2. It is clear from Tables 1 and 2 that the indium co-activator for the ZnCdS phosphor gave the maximum light output in the infrared region. Although the other ZnCdS systems activated by Au, Ga, and Cu alone have similar infrared intensities the indium activated phosphor has the greater infrared component as shown by the laser induced spectra. It is interesting to note that the white light excitation of the phosphor has an infrared output approximately 25 % than that by ultraviolet excitation this is partly due to the photons involved.

Direct Current Electroluminescent Panels

The assessment of $Zn_{0.3} Cd_{0.7} S:Cu_{0.04}$ as a possible DCEL infrared phosphor was undertaken. A number of panels were prepared with copper coated ZnCdS:Cu nitrocellulose binder. It was significant that the ZnCdS:Cu particles did not appear to surface coat with the additions used with ZnS. It is well known that the effect of Cd addition or replacement is very deleterious to both AC and DCEL performance. This was found to be the case and the Cd had a considerable effect on the surface conductivity of the phosphor.

Our belief that infrared emission is possible with AC or DCEL phosphor powder systems it was evident that a major effort is required to achieve this aim that is not achievable within the time limits of the program. It was therefore felt to be prudent to discontinue our investigations with EL and

concentrate our efforts on the light excited (PL) infrared phosphors. Clearly a range of composite systems should be prepared to be evaluated e.g. those based on mixed EL and IR powder systems or multilayered structures of IR and EL phosphors etc.

Demonstrators

Although not originally proposed we decided to construct some demonstrators using our new infrared emitting phosphors after receiving some helpful guidance from Dr. David Morton of the Army Research Labs. A number of these demonstrators were prepared for initial assessment but it must be stressed that with the limited guidance given it is still essential to know the actual requirements of the U.S. Army. The actual needs of the U.S. Army can then be met with regard to size, distance, wavelength, etc.

The initial aim was to produce a dual function matrix array mounted with both exciting (428 nm) and infrared LEDs to allow us a means of demonstrating our phosphors. We realised this type of matrix array in addition to being a source of excitation for the phosphors would allow the facility to directly transmit information either visual or covert.

The matrix array used is a robust, completely sealed unit ideal for use in a number of battlefield environments. It can be utilised by placing either a storage, anti-Stokes (upconverting) or infrared emitting phosphor panel in front of the LED array to display information; alphanumeric or graphic.

Our original agenda for the development and use of the device was that it could be used in either random or ordered sequential modes this would give a simple readily updated coding facility. A more complex version would

be developed further that would allow graphics and alphanumeric to be openly or covertly displayed.

We originally designed and built the matrix device drivers for a 'walking dot' circuit using complementary metal oxide semiconductor (CMOS) integrated circuits (IC) that was used to study and optimise on and off timings to stimulate the phosphors.

A second driver has been built with a dedicated IC that is capable of displaying characters and being fully networked for remote (distant) operation. The 64 LED matrix array emitting at 428 nm has been enabled and fully interfaced and networked for remote (distant) operation.

The ability to coat large areas with the infrared emitting phosphor to cover large area surfaces (1g could cover many cm^2 applied with a simple binder) led us to prepare laminated screens of this material.

Although not originally envisaged we developed a novel strategy to show an example of how the panels of infrared emitting phosphor could be excited at a distance. This involved interfacing a commercial EL panel to operate with an infrared remote control with a view to demonstrating how the new phosphor screens can be excited and applied. A simple laminated panel of the infrared phosphor powder may be laid over the EL panel that is used as an excitation source. Graphic information can then be conveyed by using patterned electrodes on the large area display.

A video of this set up and how it was imaged through the NVS was sent to Dr. David Morton at the Army Research Labs. Also sent was a duplicate of the laminated panel containing the infrared phosphor as seen on the video.

Conclusions and Future Work

In our assessment program we have established that a range of infrared emitting phosphors can readily be produced with promising efficiencies. Such phosphors with considerable optimisation can be sensitive to a range of excitation energies and these materials can be produced to yield cheap large area infrared displays with specific emission spectra.

Of those phosphors that have been prepared the synthesis of such systems emitting well into the infrared seem promising. It should be stressed that such phosphors have previously been overlooked and not evaluated for their night vision potential. This application points to the wide use of such systems in coding and infrared communication.

Under U.S. Government guidance we should;

a) Optimise our results to meet actual field requirements

b) Carry out detailed studies that are needed with respect to:

1) Excitation and emission efficiencies

2) Speed of excitation and response

3) Optimum lattice, activator, co-activator and flux compositions (to be refined)

4) Particle size requirements

5) Optimum combinations with other (non-IR) existing light sources such as LED and EL panels for example

We would be pleased to carry out such studies under full U.S. government control with the helpful guidance of the relevant U.S. companies.

Acknowledgements

13

Table 1

Light output of the infrared emitting phosphors measured through NVS by 366 nm excitation at 1 metre distance.

No.	Formula	Method	Flux	ApertureSize 5mm Lum. (Cd/m^2)	7mm	None
553	$Zn_{0.4} Cd_{0.6} S{:}Cu$			129	129	248
554	$Zn_{0.4} Cd_{0.6} S{:}Ag$			54	55	84
555	$Zn_{0.4} Cd_{0.6} S{:}Mn$			76	79	132
557	$Zn_{0.2} Cd_{0.8} S{:}Mn$			72	76	131
558	$Zn_{0.2} Cd_{0.8} S{:}Cu$			102	115	174
559	$Zn_{0.3} Cd_{0.7} S{:}Cu$			87	98	188
560	$Zn_{0.1} Cd_{0.9} S{:}Cu$			86	96	147
562	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.02}$	H/H		119	138	183
563	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.02}$	TDO	1%	128	145	209
565	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}$	TDO	2%	125	132	223
566	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.02}$	TDO	3%	99	129	169
569	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, Au_{0.03}$	TDO		129	146	255
571	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, Au_{0.04}$	TDO	2% NaCl	115	122	236
572	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, Au_{0.05}$	TDO	2% NaCl	116	119	235
573	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.04}, Au_{0.02}$	TDO	2% NaCl	131	132	248
574	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.02}, Au_{0.04}$	TDO	2% NaCl	123	129	240
575	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, Au_{0.04}$	TDO	2% AlCl$_3$	83	80	124
576	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}$	TDO	2% AlCl$_3$	58	64	120
577	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, In_{0.02}$	TDO	2% NaCl	128	133	221
578	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, In_{0.03}$	TDO	2% NaCl	126	98	247
579	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, In_{0.04}$	TDO	2% NaCl	124	96	261
580	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, In_{0.05}$	TDO	2% NaCl	134	84	274
583	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, In_{0.03}$	HH		58	70	141
584	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, In_{0.05}$	TDO		78	96	157
582	$Zn_{0.3} Cd_{0.7} S{:}Cu_{0.03}, Ga_{0.03}$	TDO	2% NaCl	133	126	247
585	$Zn_{0.3} Cd_{0.7} S_{0.8} Se_{0.2}{:}Cu_{0.3}, In_{0.05}$	TDO	2% NaCl	74	98	145
587	$Zn_{0.3} Cd_{0.7} S_{0.5} Se_{0.5}{:}Cu_{0.3}, In_{0.05}$	TDO	2% NaCl	24	26	51
588	$Zn_{0.3} Cd_{0.7} S_{0.25} Se_{0.75}{:}Cu_{0.3}, In_{0.05}$	TDO	2% NaCl	15	65	81
589	$Zn_{0.3} Cd_{0.7} Se{:}Cu_{0.3}, In_{0.05}$	HH	2% NaCl	27	26	26
590	$ZnS_{0.5} Se_{0.5}{:} Cu_{0.3}, In_{0.05}$	HH	2% NaCl	17	25	31
591	$ZnS_{0.7} Se_{0.3}{:} Cu_{0.3}, In_{0.05}$	HH	2% NaCl	20	24	40
592	$ZnSe{:}Cu_{0.03}, In_{0.05}$	HH		10	8	24
	ZnO			15	15	31

Table 2

Light output of the infrared emitting phosphors measured through NVS readings taken at a distance of 2 metres under 366 nm and white light excitation.

No.	Formula	Method	Flux	White Light	366 nm	*
				Lum. (Cd/m^2)		
553	$Zn_{0.4}$ $Cd_{0.6}$ S:Cu			55	215	248
554	$Zn_{0.4}$ $Cd_{0.6}$ S:Ag			11	99	84
555	$Zn_{0.4}$ $Cd_{0.6}$ S:Mn			28	121	132
557	$Zn_{0.2}$ $Cd_{0.8}$ S:Mn			20	138	131
558	$Zn_{0.2}$ $Cd_{0.8}$ S:Cu			35	153	174
559	$Zn_{0.3}$ $Cd_{0.7}$ S:Cu			37	142	188
560	$Zn_{0.1}$ $Cd_{0.9}$ S:Cu			16	141	147
562	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.02}$	H/H		40	198	183
563	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.02}$	TDO	1%	42	188	209
565	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$	TDO	2%	39	174	223
566	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.02}$	TDO	3%	40	182	169
569	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $Au_{0.03}$	TDO		47	189	255
571	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $Au_{0.04}$	TDO	2% NaCl	48	181	236
572	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $Au_{0.05}$	TDO	2% NaCl	42	175	235
573	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.04}$, $Au_{0.02}$	TDO	2% NaCl	48	200	248
574	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.02}$, $Au_{0.04}$	TDO	2% NaCl	51	208	240
575	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $Au_{0.04}$	TDO	2% $AlCl_3$	28	131	124
576	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$	TDO	2% $AlCl_3$	11	97	120
577	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $In_{0.02}$	TDO	2% NaCl	42	200	221
578	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $In_{0.03}$	TDO	2% NaCl	53	210	247
579	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $In_{0.04}$	TDO	2% NaCl	40	201	261
580	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $In_{0.05}$	TDO	2% NaCl	52	210	274
583	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $In_{0.03}$	HH		16	115	141
584	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $In_{0.05}$	TDO		22	156	157
582	$Zn_{0.3}$ $Cd_{0.7}$ S:$Cu_{0.03}$, $Ga_{0.03}$	TDO	2% NaCl	50	161	247
585	$Zn_{0.3}$ $Cd_{0.7}$ $S_{0.8}$ $Se_{0.2}$:$Cu_{0.3}$, $In_{0.05}$	TDO	2% NaCl	14	135	145
587	$Zn_{0.3}$ $Cd_{0.7}$ $S_{0.5}$ $Se_{0.5}$:$Cu_{0.3}$, $In_{0.05}$	TDO	2% NaCl	29	63	51
588	$Zn_{0.3}$ $Cd_{0.7}$ $S_{0.25}$ $Se_{0.75}$:$Cu_{0.3}$, $In_{0.05}$	TDO	2% NaCl	20	97	81
589	$Zn_{0.3}$ $Cd_{0.7}$ Se:$Cu_{0.3}$, $In_{0.05}$	HH	2% NaCl	17	26	26

*This column is the previous readings taken at 1 metre without LWPF see Table 1 for comparison

Figure 1. The spectra shows our initial results using ZnCdS doped with Cu, Mn and Ag. Excitation using 632.8 nm light.
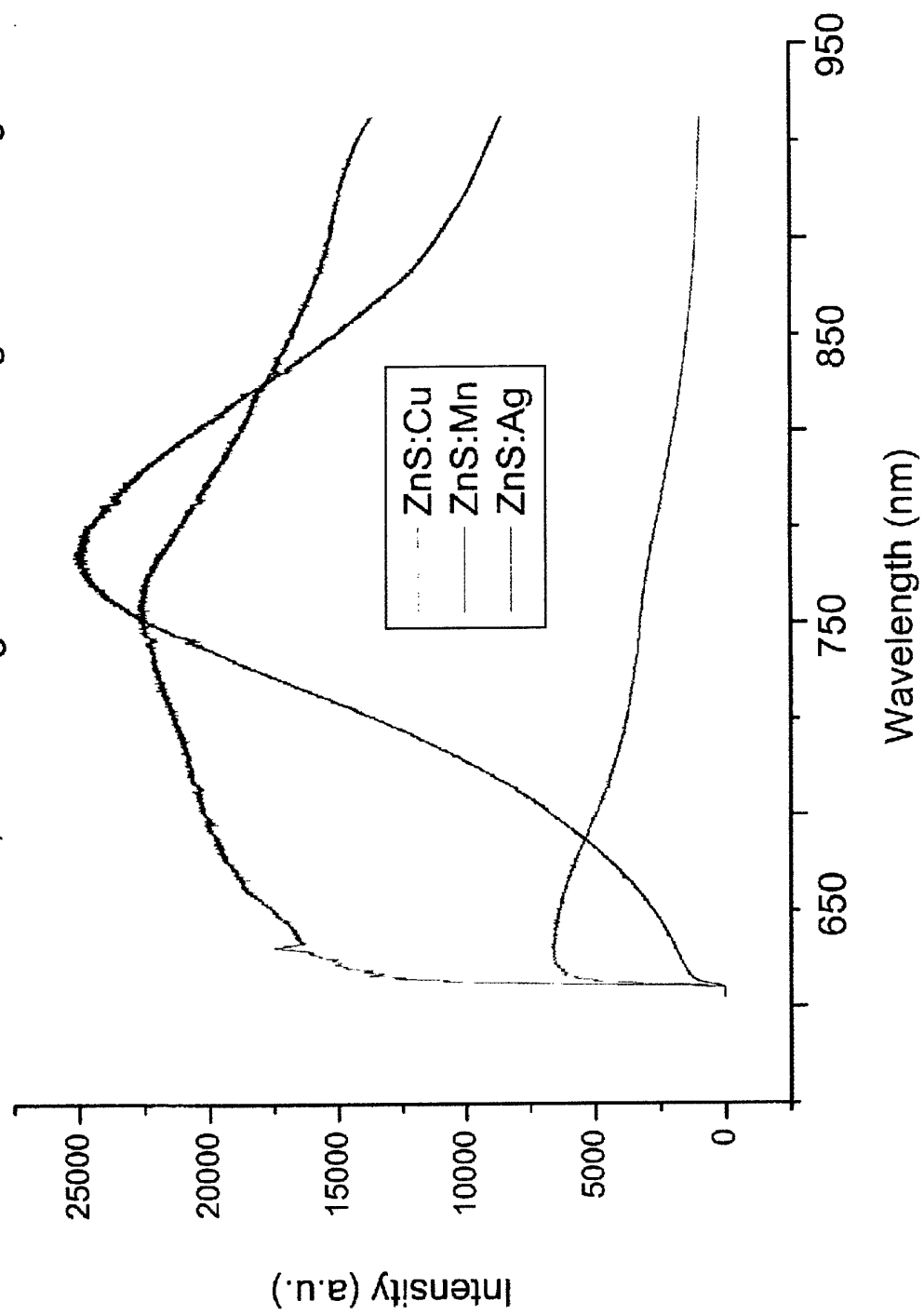
Figure 2. The spectra shows (a) the comparitive effect of Cu versus Mn as activator (b) the effect of increasing the concentration. Excitation using 632.8 nm light.

Legend:
- $Zn_{0.4}Cd_{0.6}S:Cu$
- $Zn_{0.2}Cd_{0.8}S:Cu$
- $Zn_{0.2}Cd_{0.8}S:Mn$

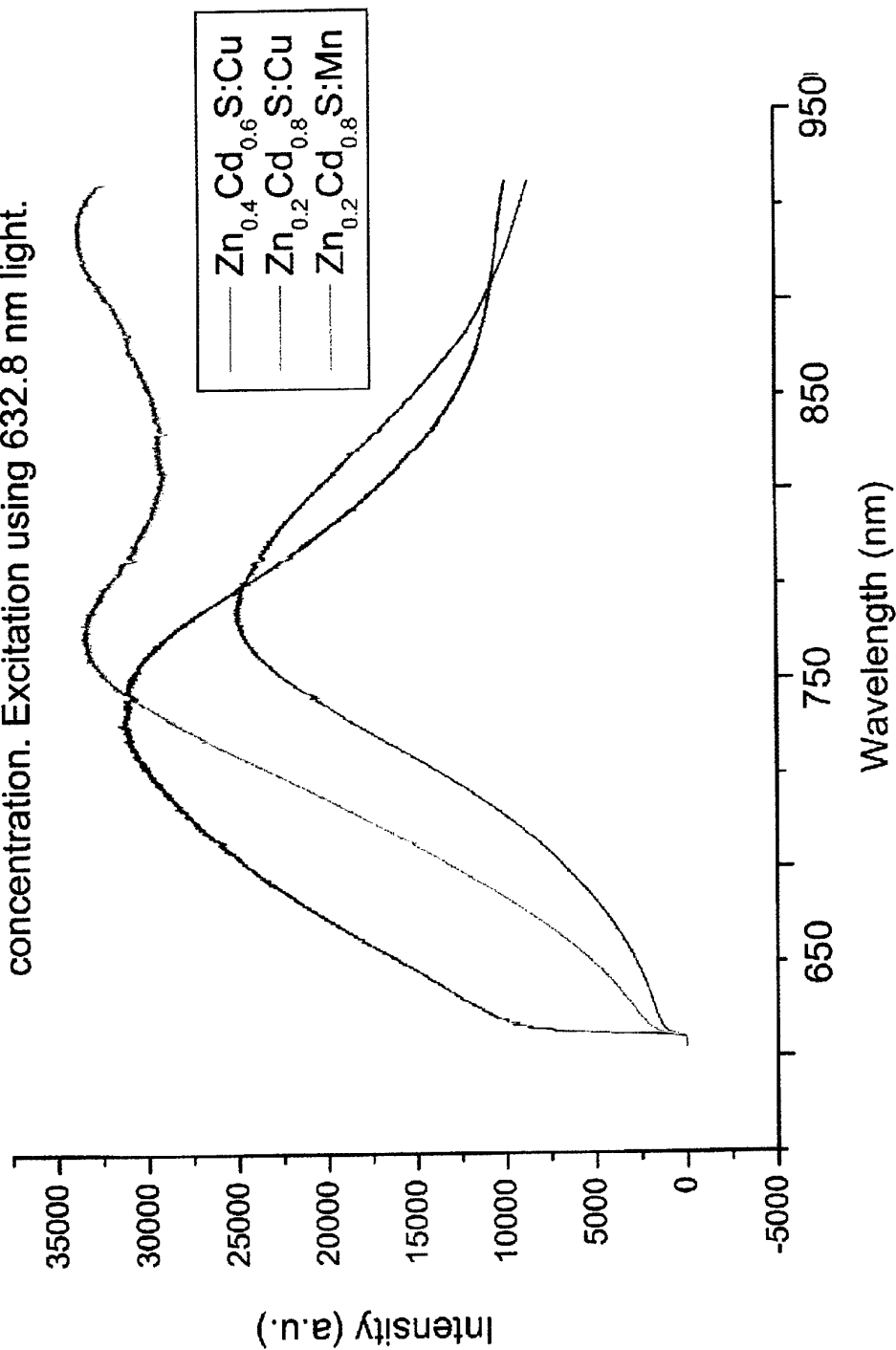X-axis: Wavelength (nm)
Y-axis: Intensity (a.u.)

Figure 3. Spectra showing the shift of the emission peak with the addition of indium as a codopant in ZnCdS:Cu replacing gold. Excitation using 632.8 nm light.

ZnCdS:Cu,Au
ZnCdS:Cu,In

intensity

wavelength/ nm